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WATER SYSTEM MICROBIAL CHECK VALVE DEVELOPMENT

FINAL REPORT

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JULY 1978

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PREPARED UNDER CONTRACT NO. NAS9-15079

BY

UMPQUA RESEARCH COMPANY

MYRTLE CREEK, OREGON

FOR

LYNDON B. JOHNSON SPACE CENTER

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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1.0 INTRODUCTION AND SUMMARY

This report describes the effort to develop and test a Residual Iodine Microbial Check Valve (RIMCV) assembly. This device is similar to the Microbial Check Valve (MCV) developed under the earlier phase of the contract with the difference that the RIMCV will cause an iodine residual to be present in the effluent water from the unit. The below described effort resulted in the development and testing of the RIMCV to an equal level as the MCV and permitted selection of the RIMCV as the optimum device to be used in the Shuttle water system.

2.0 BACKGROUND

The previous effort under contract NAS 9-15079 delivered a pre-prototype microbial check valve (MCV) assembly for use in the Shuttle potable water system. The MCV is based on an anion exchange resin that is saturated with a triiodide solution. This treatment results in essentially a zero iodine residual in the effluent water. This can be considered a deficiency in some applications since the MCV will not effect any bactericidal action in water which is stored subsequent to passing through the MCV. Included in the previous effort were (1) development tests to determine its operating characteristics; (2) design, fabrication and delivery of one preprototype unit; and (3) demonstration testing of the preprototype assembly to determine the potential use of it in Shuttle.

A resin system which is supersaturated with an iodine solution has been developed. This system causes an iodine residual to be present in the effluent water. This residual will provide continuing bactericidal action in the effluent water.

3.0 SCOPE

The scope of the effort in this phase of the contract included the development of manufacturing methodologies to make resins which produced predictable iodine residuals anywhere in the range from 1 to 20 ppm. In addition, a flight prototype design was finalized, and five prototype units were manufactured and delivered to JSC.

4.0 CONTRACTOR TASKS

The tasks required by the Contract Statement of Work are presented in this Section.

4.1 Program Plan. A program plan based on the original statement of work was prepared and submitted. Subsequently the statement of work was revised and is presented below. Tasks 4.1 through 5.0 of this report correspond to similarly numbered sections in the revised statement of work.

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EXHIBIT "B"
Revision 1
February 15, 1978

PROTOTYPE MICROBIAL CHECK VALVE DEVELOPMENT

Statement of Work

1.0 INTRODUCTION

This statement of work defines the effort required to develop and test a Residual Iodine Microbial Check Valve (RIMCV) assembly. This device is similar to the Microbial Check Valve (MCV) being developed under contract NAS 9-15079 with the difference that the RIMCV will cause an iodine residual to be present in the effluent water from the unit. The below defined effort will result in the development and testing of the RIMCV to an equal level as the MCV and will permit a selection of the optimum device to be used in the Shuttle water system.

2.0 BACKGROUND

The current effort being conducted under contract NAS 9-15079 will deliver a preprototype microbial check valve (MCV) assembly for use in the Shuttle potable water system. The MCV is based on an anion exchange resin that is saturated with a triiodide solution. This treatment results in essentially a zero iodine residual in the effluent water. This can be considered a deficiency in some applications since the MCV will not effect any bactericidal action in water which is stored subsequent to passing through the MCV. Included in the ongoing effort are (1) development tests to determine its operating characteristics; (2) design, fabrication and delivery of one preprototype unit; and (3) demonstration testing of the preprototype assembly to determine the potential use of it in Shuttle.

A resin system which is saturated with a pentiodine solution has been identified. This system causes an iodine residual to be present in the effluent water. This residual will potentially provide continuing bactericidal action in the effluent water.

3.0 SCOPE

The scope of the effort defined by this statement of work is to conduct a similar effort as being conducted under the existing contract NAS 9-15079. This effort will be conducted to determine the overall acceptability of the RIMCV for Shuttle use in lieu of the MCV. Following the completion of this effort, a decision will be made to specify which unit will be developed for the Shuttle program.

4.0 CONTRACTOR TASKS

The contractor shall perform the following tasks in the completion of this effort.

4.1 Program Plan - The contractor shall prepare a program plan to define the work they are to accomplish during the contract.

4.2 Development Testing

Research and development testing shall be conducted to produce a methodology whereby a residual iodine resin can be reliably manufactured with a specified output concentration of iodine in the range 1 to 20 ppm. In order to accomplish this objective, iodine residual as a function of throughput will be determined for resin prepared in a number of different ways including the following:

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4.2.1 Load resin with a stoichiometric I_2-KI solution. Investigate both "flow-through" and "soak" methods of loading.

4.2.2 Load resin with iodine in excess of the stoichiometric amount of I_2-KI. Investigate different loading techniques including the use of highly concentrated I_2-KI solutions as well as the successive use of less concentrated solutions.

4.2.3 Mix high residual resin with low residual resin both homogeneously and in a layered fashion.

4.2.4 Investigate other methods of loading iodine on the resin as the data obtained in tasks 4.2.1 through 4.2.3 may suggest.

4.2.5 Select the best method of formulating a high residual resin for the Shuttle application with a view to minimizing preparation time and cost while achieving a reliable and repeatable manufacturing methodology.

4.3 Refurbishing Procedures - The contractor shall develop refurbishing procedures to be used to replace the resin in the unit to make it ready for reuse.

4.4 Unit Trade Off - Following testing in paragraph 4.2, the contractor shall determine the optimum system (MCV or RIMCV) to be used in the Shuttle program. This shall include consideration of all potential use locations of the unit associated with the water system (EMU, galley, Personal Hygiene Station, and fuel cell effluent).

4.5 Preliminary Flight Hardware Documentation - The contractor shall prepare the following preliminary documentation:

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4.5.1 Contractor End Item Specification, Preliminary Certification Test

4.5.2 Plan, Preliminary

4.6 Prototype Unit Development and Testing - A prototype design shall be made following selection of the optimum unit per paragraph 4.4. A prototype unit will be fabricated and tested to demonstrate its operation.

4.7 Development of a Resin Certification Test. Develop a method of titrating the amount of iodine contained in a batch of resin.

5.0 END ITEM HARDWARE

The contractor shall fabricate and deliver five (5) units of the design selected in paragraph 4.4.

6.0 DATA

The reports defined in the attached Data Requirements List and accompanying DRDs will be required. These include the program plan, preliminary contract end item specification, preliminary certification test plan, test plan, monthly progress reports, and final report to include the program plan for developing the flight units.

Task 4.2 Development Testing

The purpose of this task was to develop a methodology whereby resins could be produced with residual iodine concentrations in the range of 1 to 20 ppm.

4.2.1 Kansas State Resins

The original intent of this program was to evaluate a "penta iodide" or high residual iodine resin that was reportedly available from the inventors of the basic resin at Kansas State University (KSU). Initial efforts at KSU produced a high residual resin, however it washed out to produce essentially the same results as their low residual resin as shown in Figure 4-1. The solid squares represent the KSU "penta" resin, the solid circles represent resin made commercially by Water Pollution Control, Inc. and the solid triangles are the KSU "tri"-iodide or low residue resin. These data were reportedly obtained at KSU on 1.91 cm dia x 15.24 cm long beds at 0.25 liters/min. This produced a residence time of 1.57 seconds compared to 2.0 seconds for the prototype configuration. The mission man-day scale (see Figure 4-1A) shows an unacceptable range of iodine residual for both the high and low residual KSU resins and the commercially produced resin over the 210 man-day equivalent range for a 7 man crew for 30 days. Consequently, the program plan was modified to initiate an effort to produce a high residual resin.

4.2.2 Resin Development

The low residual, tri-iodide resins produced at KSU contain approximately 97% of the theoretical exchange capacity of the ion exchange resin as I_3^-. Consequently, various methods were investigated to introduce more iodine into the resin. The mechanism used by KSU is intended to produce a I_5^- complex which only contains iodine at the exchange sites of the resin. However, it is not clear what the actual state of the iodine is once the amount necessary for stoichiometric occupation of all sites by I_3^- is exceeded. Some form of solution of I_2 in the resin matrix is also possible.

A series of resin batches was manufactured in order to obtain a resin which introduced iodine in the 2-5 ppm range. This was accomplished by first converting the raw Ionac 540 (or 544 which is food

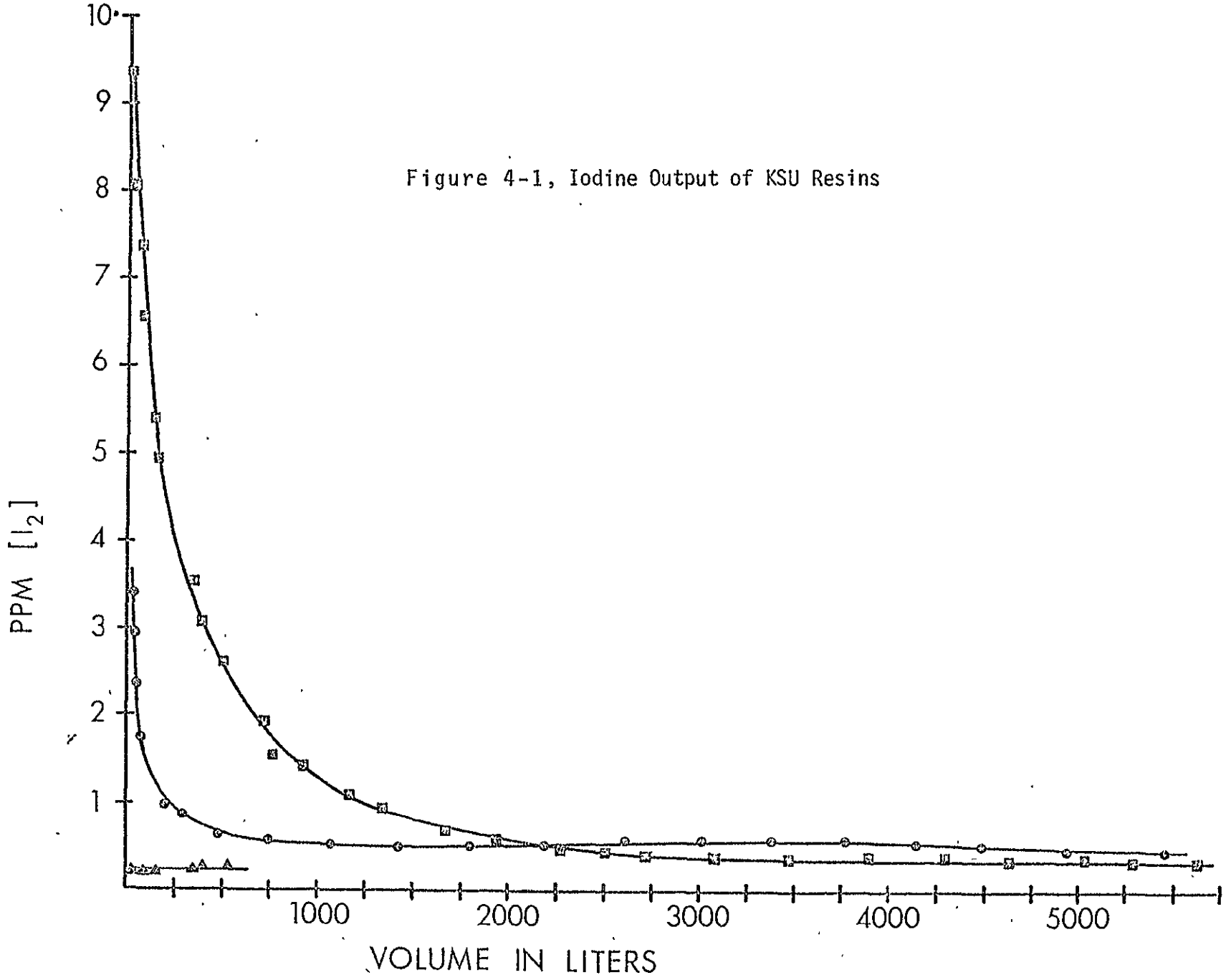

Figure 4-1, Iodine Output of KSU Resins

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grade) from the chloride form to the iodide form by soaking in a 1N solution of KI which contained 3.5 times the number of equivalents of I^- based on the theoretical capacity of the resin. Various strengths and methods of treating the resin with I_3^- solutions were then investigated, the most significant of which are described below. The method of resin preparation finally selected for the Shuttle is presented in Appendix A. This method produces a 2 ppm residual with a nearly flat rinse out characteristic as shown in Figure 4-1A.

Batch 70925

Pretreated resin was soaked in a 1N solution of I_3^- containing 132% of the stoichiometric amount of I_2. The mixture was stirred for 3 hours, and allowed to continue soaking overnight. After rinsing, a bed was tested at a flow rate equivalent to 0.45 kg/min (1 lb/min). The initial iodine residual was 1.5 ppm, and fell to <0.2 ppm after 50 hours. At this time a liter of E. coli cells at 10^6/ml was passed through the bed. Less than 10/ml were found in the effluent, thus confirming biocidal activity at less than 0.2 ppm I_2, as is reported for the KSU low residual resin.

Batch 71128

Pretreated resin was soaked in a solution of I_3^- which contained 125% of stoichiometric. After soaking, the decanted solution was titrated, and the resin found to have absorbed 118% of its theoretical exchange capacity (based on I_3^-). The resin was then soaked in a fresh solution containing 125% of theoretical. The resin absorbed an additional 103% of theoretical capacity. A third soak in solution containing 180% of the resin's theoretical capacity added another 7%. At this point it was assumed that the resin was saturated. The total amount of iodine absorbed was equivalent to 228% of its theoretical exchange capacity. If it is absorbed in a I_5^- form on active sites only, the theoretical capacity would be only 200% of the I_3^- capacity. This indicates that some iodine is absorbed in a manner not involving active sites unless there is a stable I_n^- with n greater than 5. After draining, the resin was tested and found to produce 45 ppm residual iodine initially, dropping to 20 ppm at 20 hours, 5 ppm at 100 hours, 1 ppm at 250 hours and 0.2 ppm at 500 hours.

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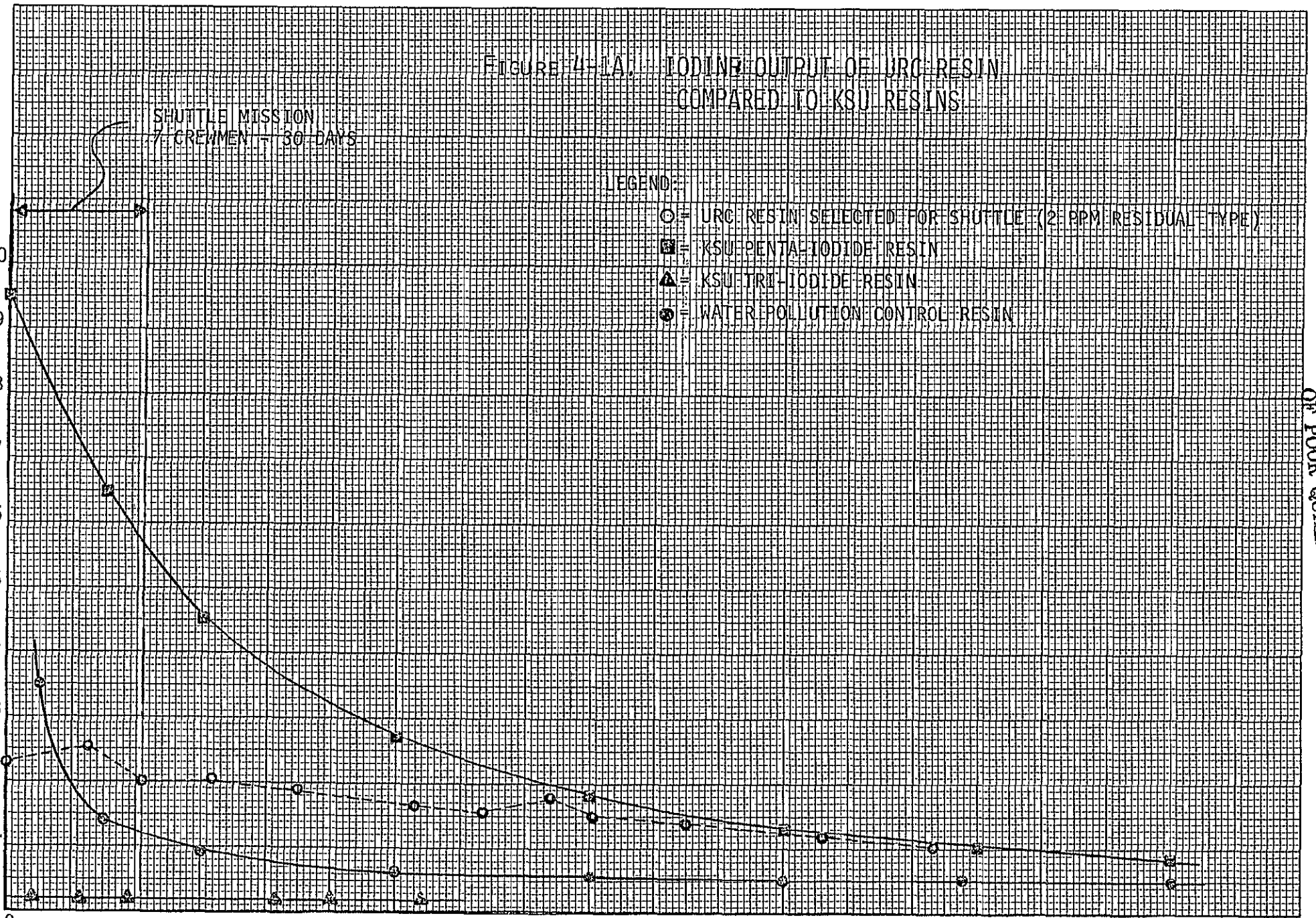
FIGURE 4-1A. IODINE OUTPUT OF URC RESIN COMPARED TO KSU RESINS



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tests. A bed was prepared by placing a layer of low residual resin followed by an equal quantity of the high residual resin as prepared above. The second bed contained an intimate mixture of equal parts of the two resins. The test results are shown in Figure 4-2 . The shapes of the two curves indicate that by proper combinations of mixing and layering, beds with stable and predictable washthrough characteristics may be manufactured.

Batch 80105

Pretreated resin was soaked in 158% stoichiometric solution, absorbing 144% of theoretical.Initial iodine residual was 4 ppm, washing down to 1 ppm after 500 hours at .23 kg/min (1/2 lb/min) which approximates the peak fuel cell flow.

Batch 80118A

A 158% solution was poured through a bed of pretreated resin. After rinsing the iodine residual never reached 0.2 ppm, indicating that the pourthrough technique is not an effective method of resin preparation.

Batch 80118B

A portion of 80118A was then soaked in a fresh solution containing 158% of stoichiometric. This combined procedure introduced 254% iodine into the resin. Flow testing showed initial residual iodine at 8 ppm, reducing to 5 ppm at 200 hours.

The following sub batches are different post treatments of the first batch.

Batch 80224A

A 240% stoichiometric solution was poured through pretreated resin, resulting in 187% absorption. This resin was the basis of the rest of the 80224 series. Iodine output is shown in Figure 4-3.

Batch 80224B

Part of 80224A was allowed to soak in the solution after the initial pour through, yielding a total of 218% uptake.

Batch 80224C

A portion of 80224A was soaked in a 120% stoichiometric solution, resulting in a total 278% uptake.

Batch 80224D

Part of 80224A was soaked in 240% stoichiometric solution, yielding 326% uptake.

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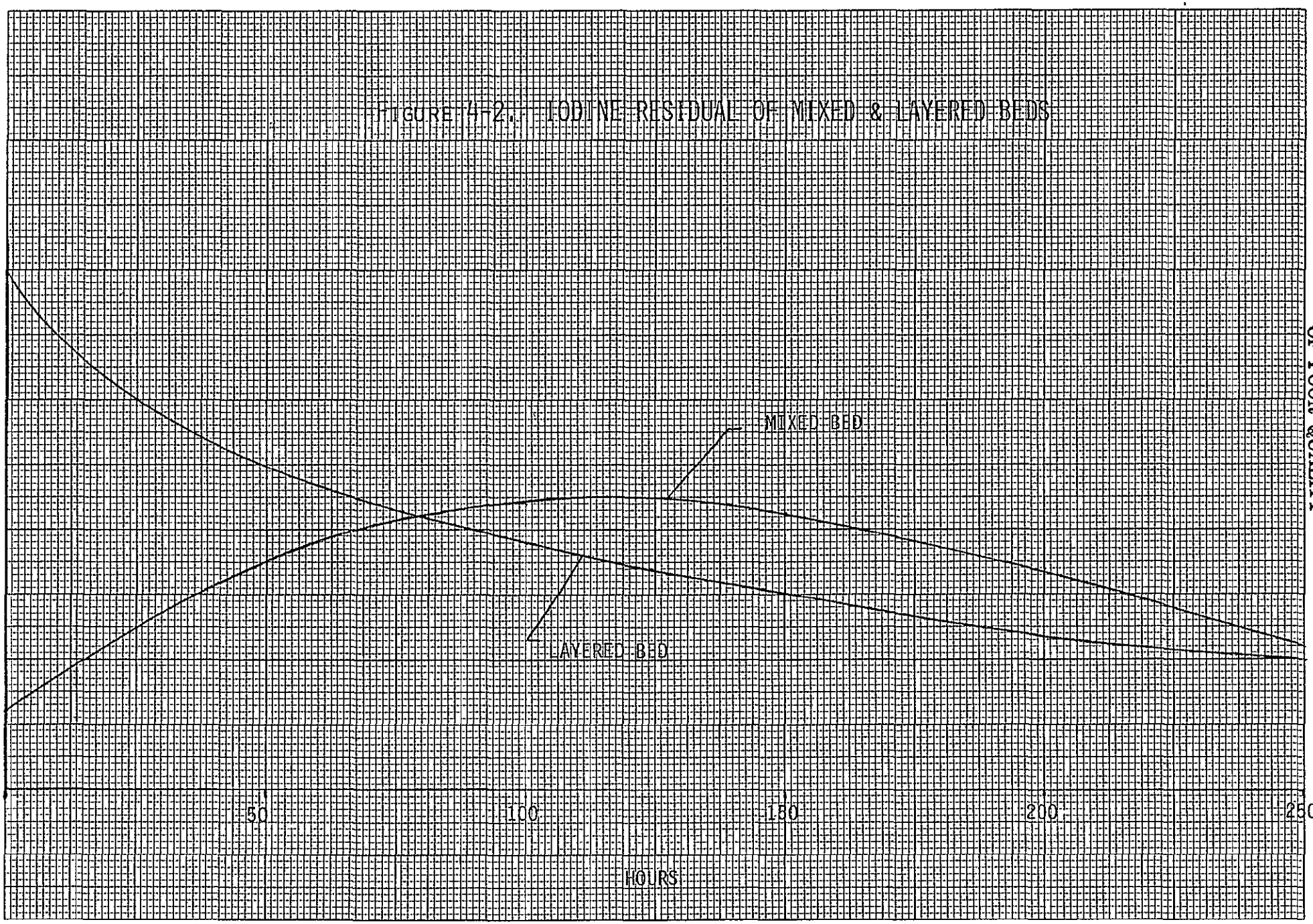
FIGURE 4-2. IODINE RESIDUAL OF MIXED & LAYERED BEDS



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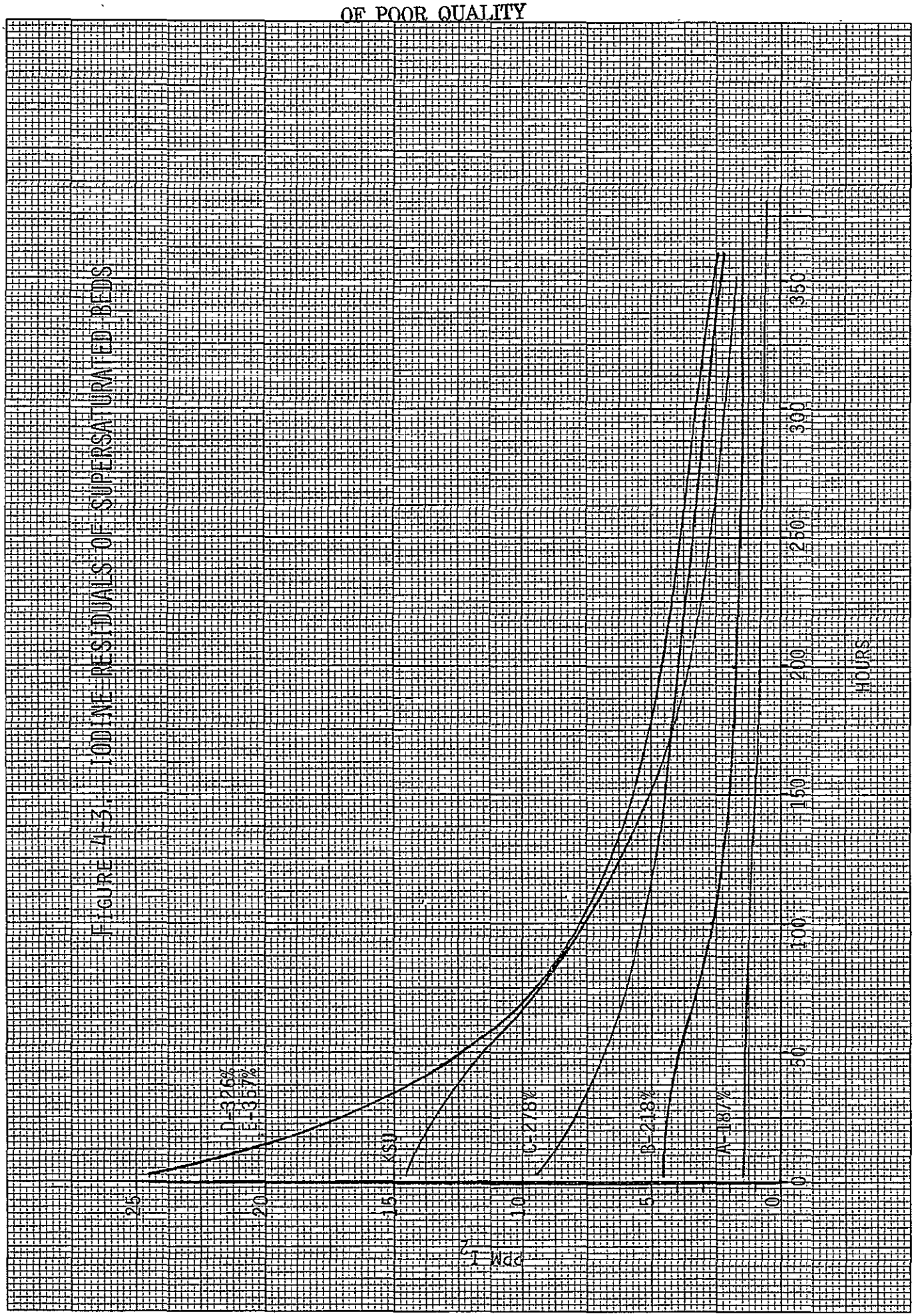

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Batch 80224E

Part of 80224A was soaked in 360% stiochiometric solution, resulting in 357% uptake.

The iodine residual curves for Batches D and E were essentially indistinguishable, and are quite close to that of a "penta" iodide resin obtained from KSU.

The following Batches were attempts to zero in on a resin to yield a 2-3 ppm resin.

Batch 80316

Pretreated resin soaked in 145% stoichiometric solution, absorbed 142% of theoretical. The residual iodine stayed between 1.4 and 1.5 ppm for over 100 hours, but still somewhat lower than desired.

The following resins were prepared from a new 45 kg lot of Ionac 544.

Batch 80403

Pretreated resin soaked in 170% stoichiometric solution, absorbing 168% of theoretical. Iodine residual remained between 3 and 4 ppm after 200 hours.

Batch 80410

Pretreated resin soaked in 163% stoichiometric solution, yielding 151% absorption. Iodine residual started at 8 ppm but washed to 2 ppm at 100 hours. Apparently this resin was not adequately rinsed before drying.

Batch 80501

This was the first attempt at a large, 1 kg, batch of resin. Pretreated resin soaked in 170% stoichiometric solution, yielding 161% absorption. Iodine residual started at 3.5 ppm and washed out to 1.3 ppm at 200 hours.

Batch 80511

This also was a large batch. Soaking was carried out the same as 80501. The initial iodine residual was 2.5 ppm and followed the curve of 80501.

It is apparent that closer control of the rinsing process before drying is necessary in order to closely achieve the initial iodine residual. However the basic methodology has been developed to manufacture a reasonably predictable high residual resin.

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4.2.3 Additional Testing

Two tests were conducted on the resin to verify previously reported performance limitations and insure compatibility with the Shuttle water system loading procedure.

Spore Challenge

Limited testing during the development of the "tri" iodide resin at KSU indicated that it was not effective against spores.

A resin that had a 2 ppm iodine residual was tested with a suspension of Difco Bacillus subtilis spores at room temperature. Since the usual procedure used to produce spores is by heat shocking the cells, samples of the influent spore suspension and resin bed effluent were split, 1/2 used for a control, the other 1/2 heat shocked for 10 minutes at $90^{o}C$. Each sample was plated in triplicate on Standard Methods plate count agar and Difco Antibody medium #1. (See Figure 4-4). The bed was challenged with 1.0×10^{5} spores/ml. The heat shocked effluent contained less than 10 organisms/ml while the control effluent contained 1.3×10^{5} organisms/ml. This indicates that the resin had little or no effect upon the viability of the Bacillus subtilis spores, but they were effectively devitalized by approximately 2 mg/l iodine at $90^{o}C$ for 10 minutes.

Vacuum Exposure

The Shuttle water system will be loaded by completely evacuating the system and backfilling water.

The effect of vacuum was investigated by exposing a bed of resin to 3 KPa (1 in Hg) for 6 hours at room temperature. No significant weight loss was measured. The bed was then tested together with an identical bed from the same batch for 200 hours. No significant difference in iodine residual was indicated.

Materials Testing

Metal alloy samples provided by JSC were immersed in wet resin for 15 months. The water was then diluted to 25 ml and tested for the major constituents of the alloys by atomic absorption spectrophotometry. No visible corrosion was evident on any of the samples. The water in the Inconel 825 had a visible red color.

The most suitable alloy appears to be 316L although both 321 and 347 would be satisfactory. The unavailability of 316L in standard tubing sizes would severly impact the price and schedule of flight units. Therefore 316 stainless steel was selected. In addition, 316 prototype units have shown no corrosion problems.

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Table 4-1, Metal Ions Leached from Alloys
(Complete immersion in wet resin, 15 months vented)

	PPM METAL ION IN 25 ML H_2O/GM				
	Mn	Cu	Fe	Cr	Ni
321	<0.01	0.03	0.25	<0.02	<0.05
HWT	<0.01	<0.01	<0.07	<0.02	<0.05
316L	<0.01	<0.01	<0.07	<0.02	<0.05
Hastalloy Alloy G.	<0.01	0.01	0.36	<0.02	<0.05
Alloy 6X	<0.01	0.02	0.42	<0.02	<0.05
Inconel 600	<0.01	0.12	4.5	0.08	4.5
Inconel 718	<0.01	0.03	0.14	<0.02	<0.05
Alloy 29-4	<0.01	<0.01	<0.07	<0.02	<0.05
347	0.06	0.03	0.29	<0.02	<0.05
Inconel 825	0.06	0.23	5.1	<0.02	44.7
Inconel 625	<0.01	<0.01	0.99	<0.02	<0.05
Resin Control	<0.01	0.01	0.39	<0.02	<0.05

Figure 4-4. SPORE CHALLENGE SCHEMATIC

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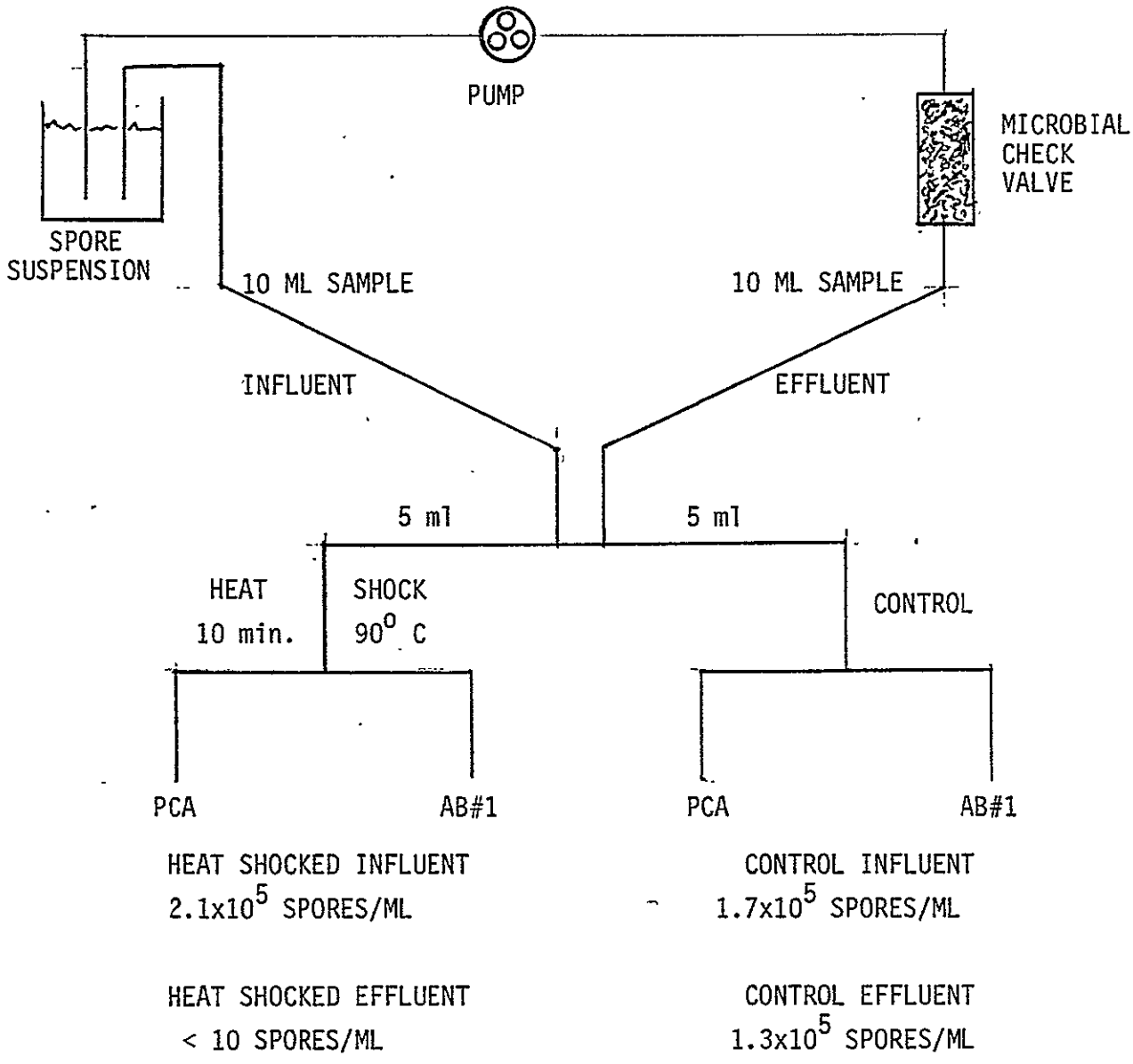


4.3 CHECK VALVE REFURBISHING PROCEDURE

The following stepwise procedure has been developed which is used in replacing the resin in a previously used (or new) MCV.

Place Check Valve in holding fixture with retaining ring end in the up position. Using compression fixture, depress end below snap ring, which will compress pressure spring, inside body and remove retaining ring.

Discard retaining ring.

Release compression fixture and allow end cap to extend to its free position. Remove compression fixture. Remove end cap, spring, screen assembly and cup seal. Remove O-ring seal from end cap and discard O-ring seal.

Remove valve from holding fixture and using a suitable container empty resin bed into it. Insert screen tool into small end of valve and secure into threaded center of screen assembly. Screen assembly can now be pushed out through open end of valve. Remove screen assembly from holding tool. Remove cup seal from small end of valve taking care not to damage seal.

Remove fittings from end cap and small end of valve body. Remove O-rings seals and discard seals. Thoroughly wash all parts in clean water. Examine all parts for damage, wear, corrosion or foreign matter.

In a clean facility, thoroughly clean and wash all parts in alcohol solution, making sure all inside corners and retaining ring groove are clean.

Rinse in D.I. water and allow to air dry. Apply O-ring grease. Replace O-ring seals onto end fittings and onto end cap.

Insert screen holding rod through small end of valve body through one cup seal and into screen assembly. (Lip of cup seal should be facing away from large end of valve). Center screen assembly into cup seal and position inside valve body on shoulders at base of valve body. Install valve with screen holding tool into holding

4.3 (cont) fixture with large open end of valve in the up position.

Pour pre-measured amount of resin into valve using funnel.

Note: Hold funnel as close as possible to screen assembly in valve body to eliminate resin bounce.

Position second screen assembly into relief in cup seal with fine screen resting on cup seal flange. Insert screen assembly with cup seal into large end of valve and position over resin using placement tool. Remove placement tool and insert compression screen assembly over locating ring on back of screen assembly.

Position end cap onto compression spring making sure spring is located inside groove in bottom of end cap.

Using compression fixture compress end cap into valve just far enough to install retaining ring.

Remove compression fixture and screen holding tool. Install Fittings into valve end and tighten to TBD in-lb torque.

4.4 UNIT TRADE OFF

Two configurations at the MCV have been demonstrated. The original MCV concept incorporates a resin which introduces minimum residual iodine into the water, (less than 0.2 ppm), while maintaining the capability of effectively preventing the passage of viable microorganisms. The RIMCV concept introduces iodine into the water at predeterminable levels (2-5 ppm) thus providing residual biocidal capability as well as preventing the passage of viable microorganisms.

Potential use locations of the unit include the EMU recharge and dump lines, galley dispensers, the personal hygiene station, and the fuel cell effluent line. With the exception of the fuel cell effluent line, all of the above locations terminate in an exposed port which is succeptable to contamination and consequently must have a means to prevent back contamination of the rest of the potable water system. Both MCV configurations have demonstrated

4.4 (cont) the ability to meet this requirement. The original Shuttle Water System design included a Silver-Ion Generator to provide a residual biocide in the potable water system. Difficulties with the development of this system led to the decision to drop the Silver-Ion Generator.

Consequently the RIMCV which introduces iodine into the water is exceptionally suitabe for the fuel cell effluent line application. The use of this configuration also guarantees residual biocide at each use point, even if degredation occurs in the storage and distribution systems.

In view of the ability of the RIMCV to effectively act as a biocide source as well as a microbial check valve, it is clearly the most advatageous configuration for the intended Space Shuttle applications.

4.5 Preliminary Flight Hardware Documentation

A preliminary Contract End Item Specification and Preliminary Certification Test Plan for the Flight Hardware program are included under separate cover.

4.6 PROTOTYPE UNIT DEVELOPMENT AND TESTING

A microbial Check Valve Prototype was designed and fabricated. Pressure drop data were obtained, and a 30-day simulated mission test was conducted.

4.6.1 Prototype Design. The final prototype design was based on the preliminary design developed during the earlier phase of the contract and test experience during this phase. The cartridge design is shown in Figure 4-5 and is fully described in three "C" size as-built drawings number NAS9-15079 and titled: "Microbial Check Valve Assembly" sheets 1, 2 and 3. The body consists of a section of 316 Stainless Steel Tube with the end and retainer collar welded on. The resin (16 to 50 mesh) is contained between two perforated SS[1] discs which are faced with 100 mesh SS[1] screens. During loading tests it was determined that the screens should be attached to the discs, and those in the prototype units were epoxyed. The flight units will have the screens attached by spot welding. A spring centering ring is tack-welded to the backs of the discs. Early versions of the prototype had the discs closely fitting the inside of the body, however the finer resin particles began to appear in the effluent stream. The discs were then ground smaller and cup seals were machined from Teflon to provide a positive seal along the walls of the housing. This eliminated the loss of resin particles.

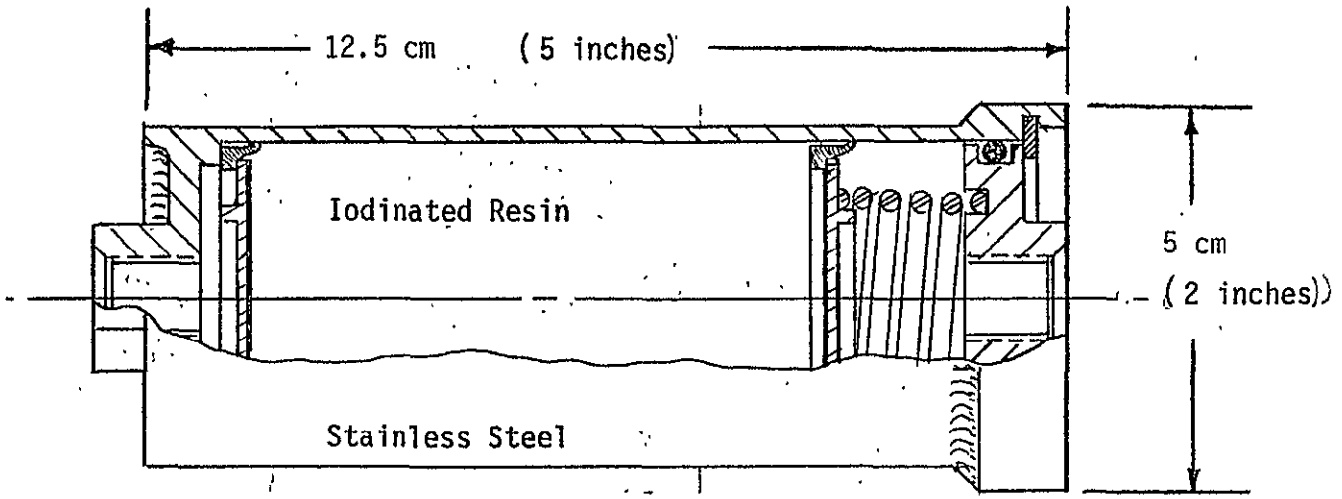
The resin compression spring seats on the back of the upper disc, and is compressed by the end plug which is retained by a snap ring. Both end ports are threaded to accept AN fittings. The plug is sealed with Vitron O-ring.

4.6.2 30-Day Simulated Mission Test. Two MCV prototype units were tested for 30 plus days under conditions designed to simulate Space Shuttle useage. During the test microbial profiles were taken four times and a complete analysis of water quality was performed near the end of the test. The following parameters were monitored on a continuous basis: pressure drop, iodine residual, specific conductance and pH. These data are presented in the following paragraphs.

4.6.2.1 Test Set-up. A schematic of the apparatus used to simulate the expected load of a 30 day mission is presented in Figure 4-6. City tap water was

1. SS = Stainless steel (18-8 Series)

Figure 4-5, MICROBIAL CHECK VALVE PROTOTYPE



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fed through a deionization bed, deaerated in a heated air trap then supplied on demand to a glass lined water tank via a float switch controlled solenoid valve. The water was gravity fed from the water tank to two resin filled Microbial Check Valve (MCV) cartridges through timer activated solenoid valves. The flow through each bed was controlled by manual adjustment of the height of the MCV outlet in relation to the fixed head of water maintained in the water tank by the float switch.

In the initial days of the test run; water in the system was highly aerated from the installation of a fresh deionization cartridge. The air became trapped in the resin beds causing an increase in the pressure drop across the MCV unit. This problem was alleviated by the installation of an air trap upstream of the water tank.

Figure 4-6. 30-DAY TEST SETUP

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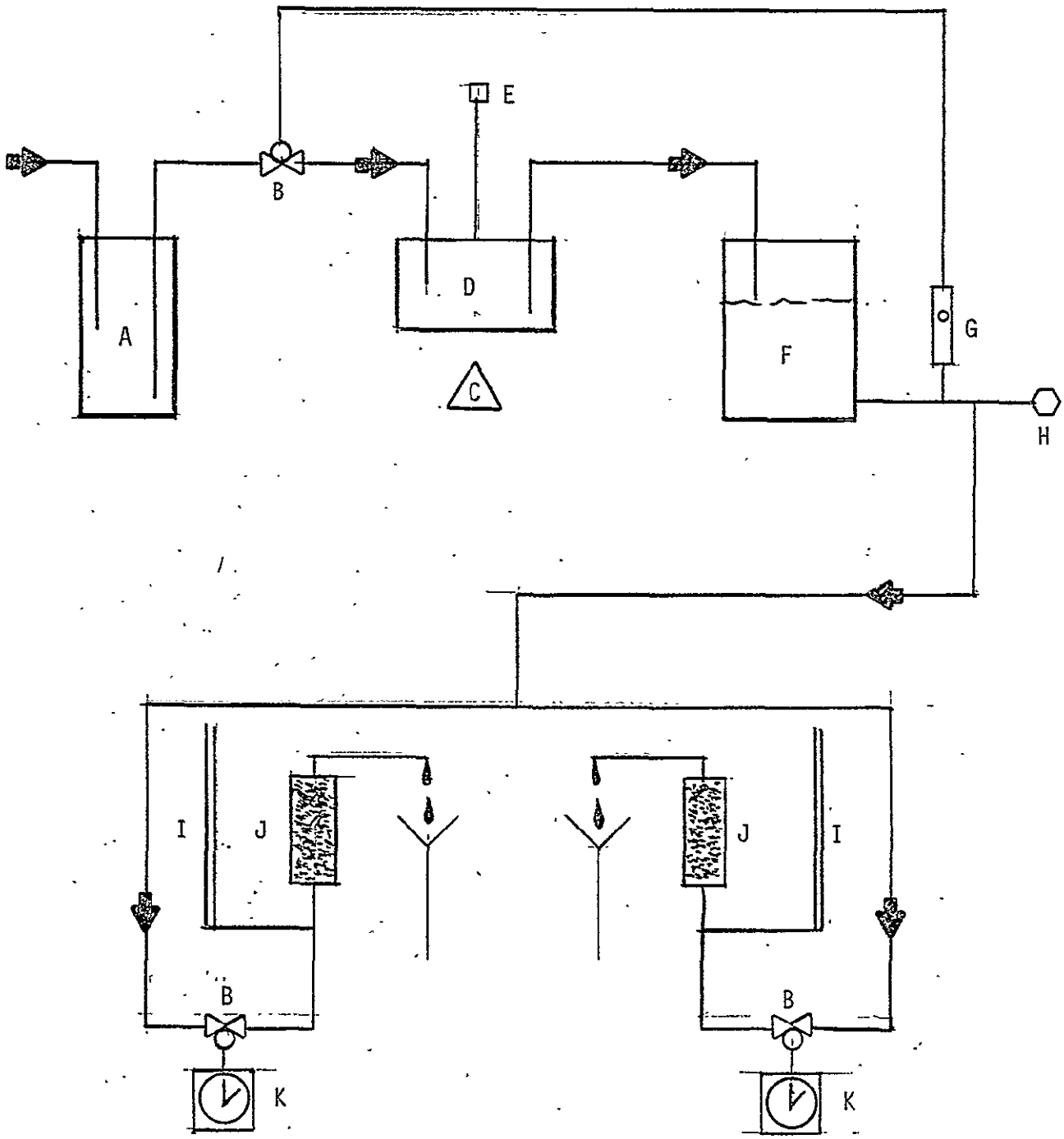

A. WATER DEIONIZER
B. SOLENOID VALUE
C. HEATER
D. AIR TRAP
E. AIR VENT
F. WATER RESERVOIR
G. FLOAT SWITCH
H. INFLUENT SAMPLE PORT
I. MANOMETER
J. MICROBIAL CHECK VALVE
K. TIME SWITCH

Flow Profiles. The MCV has two basic flow modes associated with its planned use in the Space Shuttle program: (1) the fuel cell effluent to the potable water system will be essentially continuous with flow rates ranging from 5.5 kg/hr to 10 kg/hr; and (2) the various water dispensing points all require short, relatively high flow rate performance, i.e., 0.5 kg/min for 1 minute or less. In order to simulate these conditions, two prototype MCV units, MCV#P6 and MCV#P7 were tested for 30 + days with Batch 80511 Resin using the following flow profiles:

MCV #P6 FLOW PROFILE
(SIMULATED FUEL CELL FLOW)

FLOW KG/HR	TIME DURATION HR	FREQUENCY DAY^{-1}	TOTAL FLOW KG/DAY
10	1/2	2	10.0
5.4	1	1	5.4
5.4	3	1	16.3
			31.7

MCV #P7 FLOW PROFILE
(SIMULATED DISPENSER FLOW)

FLOW KG/MIN	TIME DURATION MIN	FREQUENCY HR^{-1}	TOTAL FLOW KG/DAY
0.45	1	1	10.9

4.6.2.3 Microbial Data. Standard 48-hour aerobic plate counts were obtained on the influent and effluent four times during the 30-Day Test. The results are presented below.

AEROBIC 48-HR PLATE COUNTS
(NUMBER OF ORGANISMS PER ML)

	Elapsed Time			
	26 hr	193 hr	506 hr	674 hr
INFLUENT	3.2×10^5	3.1×10^4	8.8×10^4	3.6×10^4
EFFLUENT MCV #P6	<10	<10	<10	<10
EFFLUENT MCV #P7	<10	<10	<10	<10

An input level of 10^4 to 10^5 organisms per ml was maintained throughout the test. The output level was always less than 10 organisms per

ml. These organisms were indiginous to the laboratory deionized water system, and are probably typical of what will accumulate in any closed system without a residual biocide.

4.6.2.4 Pressure Drop Investigations. Pressure drop investigations were conducted using a test setup similar to that described in the Interim Report. Springloads of up to 20 pounds were investigated with a special weight-loaded bed (See Figure 4-7) and found to have no short term or long term effects on pressure drop. The time related increase in pressure drop, which was observed in previous testing, was found to be related to the amount of entrained gas in the challenge water. It appears that entrained gas is removed by the resin bed and tends to block-off flow passages resulting in an increase in pressure drop. This phenomenon was especially noted after installation of a newly recharged commercial deionizer in the laboratory water conditioning system, which produced water heavily charged with micro-bubbles for several days after installation. During this period the pressure drop in the MCV beds rose at about 10% per day (See Figures 4-8 and 4-9). At the 50-hour mark, the gas that had accumulated in the beds was removed with an aspirator, which lowered ΔP by 15 to 20%. At the 90-hour mark a deaerator was installed in the feed water circuit and pressure drop declined over the next few days to the levels shown in Figures 4-8 and 4-9, which are about 1/3 of the design point maximum.

Figure 4-7. APPARATUS USED FOR INVESTIGATING THE EFFECT OF SPRING LOADING ON PRESSURE DROP.

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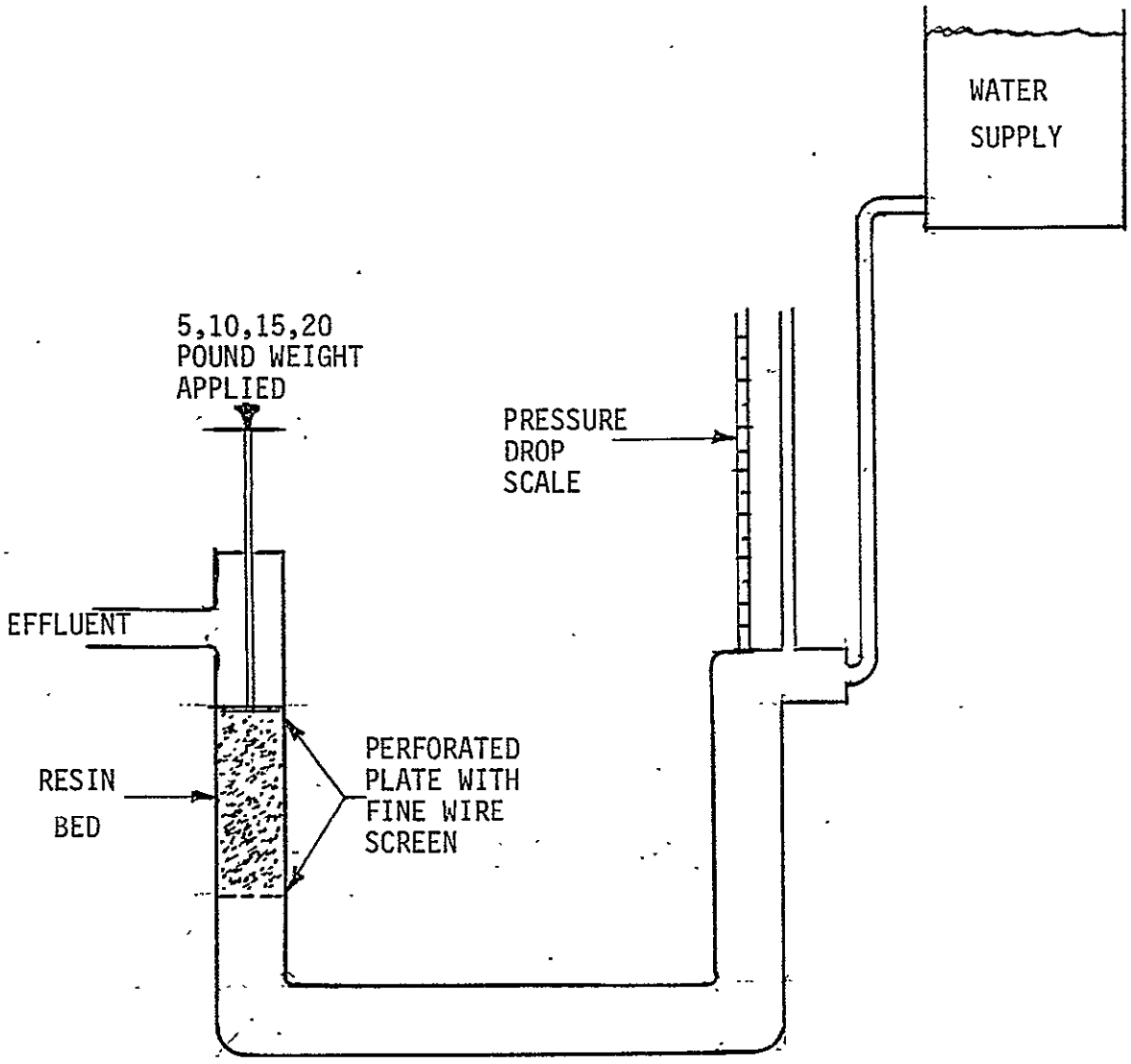

The pressure drop in a resin bed is described as follows:

$$\Delta P = K \frac{WL\mu}{d^2}$$

where: ΔP = pressure drop, psi

W = flow, lb/min

L = bed length, in

d = bed diameter, in

μ = water viscosity, centipoise

Design values for the MCV are:

ΔP = 0.5 psi

W = 22.8 lb/hr (0.38 lb/min)

L = 3 in

d = 1.62 in

μ = 1.0 at 68.4°F

Therefore, the maximum allowable value of K is:

$$K_{max} = \frac{\Delta P d^2}{WL\mu} = \frac{(0.5)(1.62)^2}{(0.38)\ (3)(1.0)}$$

$$= 1.15$$

These pressure drop tests were run well in excess of 30 days. The K values stabilized between 0.32 and 0.45 in both tests, which is approximately 1/3 of the maximum allowable value.

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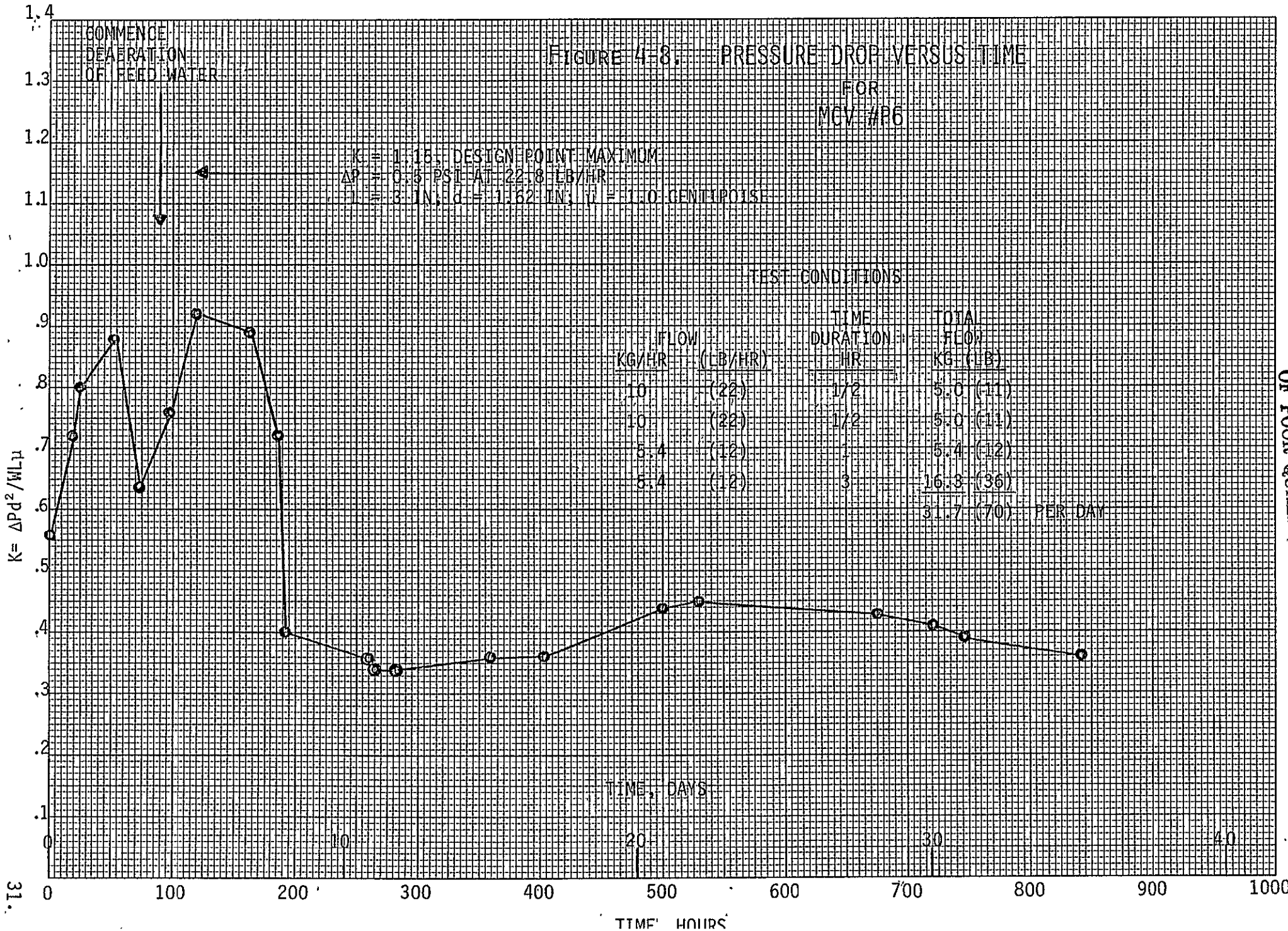

FIGURE 4-8. PRESSURE DROP VERSUS TIME FOR MCV #P6

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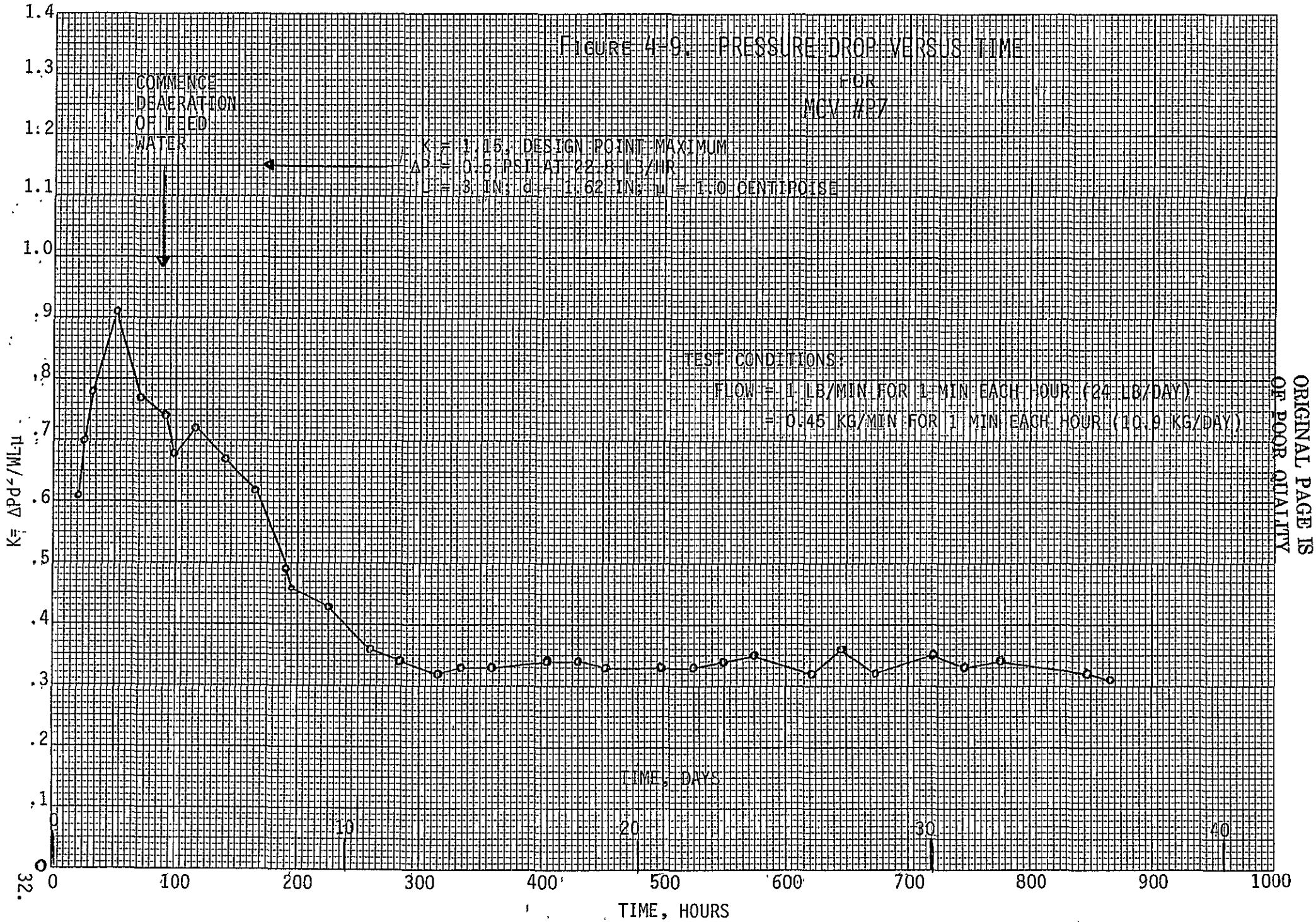

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32.

4.6.2.5 Iodine Residual. The iodine residual data are presented in Figures 4-10 and 4-11. The design goal for iodine residual is 2 to 3 mg/l. This goal was achieved during the first 90 hours of operation with aereated water. But after the commencement of deaereation the iodine residual increased to the 2.5 to 4 mg/l range. This is attributed to the elimination of trapped air in the resin bed which would tend to impede the mass transfer of iodine. During the time interval of 90 to 400 hours the average iodine residual exhibited a gradual decline for both units. This is expected behavior. MCV #6 had approximately 0.5 mg/l higher iodine residual than MCV #7. This is possibly due to the lower flow rates of MCV #6. After 400 hours the iodine residual from both valves increased to the 3.5 to 4.5 mg/l range. Since this increase occurred to both units at the same time and the two units had significantly different flow rates and total throughput, it is felt that the increase was a result of an external factor not related to flow or throughput. Temperature change, iodine test reagents and organic load change were all examined and ruled out as potential causes of the increase. The increase is apparently the result of some alteration in the feed water characteristics, but at this time the cause has not been identified.

It is expected that the resin for the MCV flight units will be prepared with an appropriately lower initial iodine residual.

4.6.2.6 Specific Conductance. The effluent specific conductance was almost identical for both MCV units. The data are presented in Figures 4-12 and 4-13. Initial values were 40 μ siemens/cm with a logarithmic decline to around 3 μ siemens/cm. After 30 days a small unexplained increase of 1 to 2 μ siemens/cm was noted.

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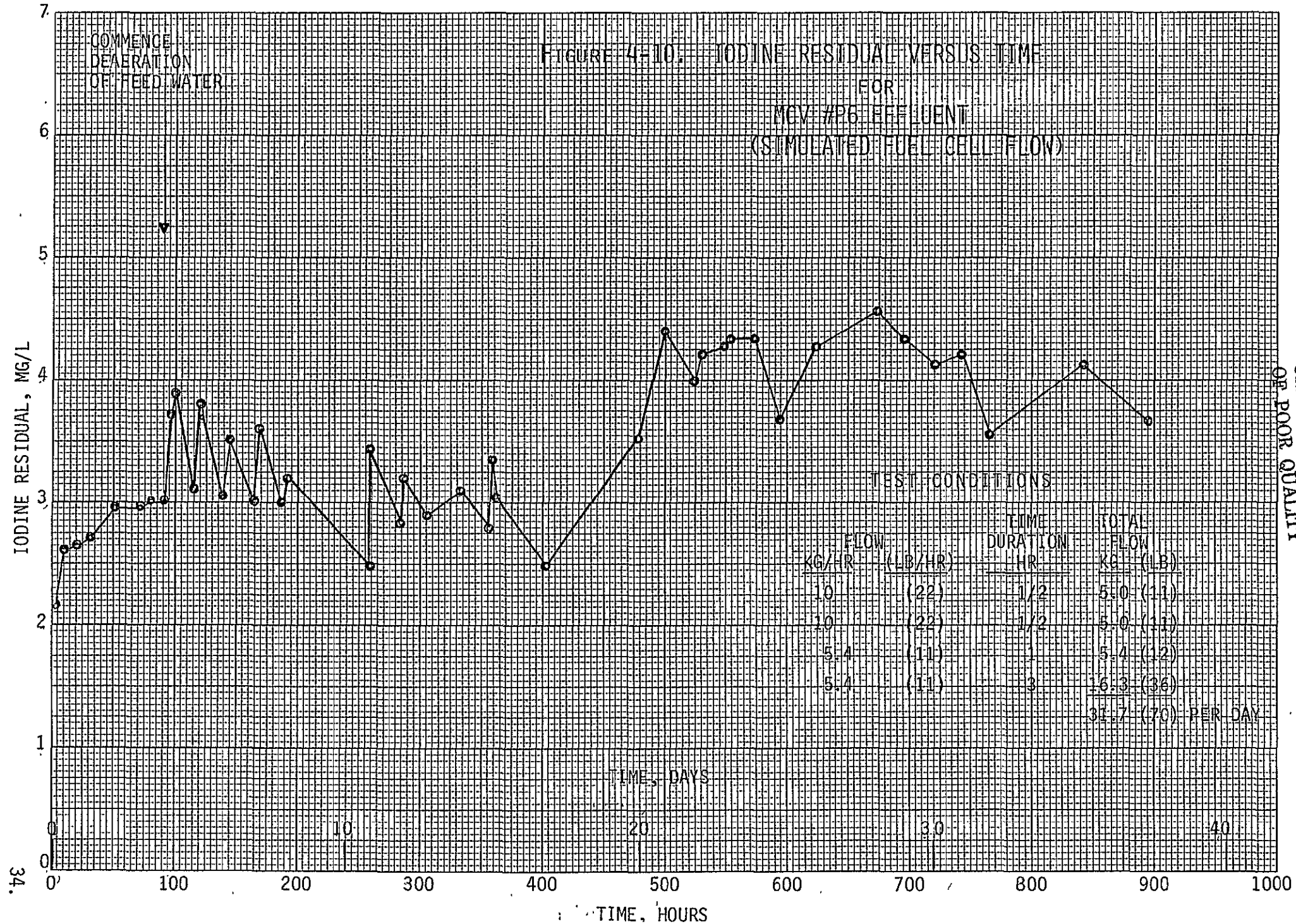

FIGURE 4-10. IODINE RESIDUAL VERSUS TIME FOR MCV #P6 EFFLUENT (SIMULATED FUEL CELL FLOW)

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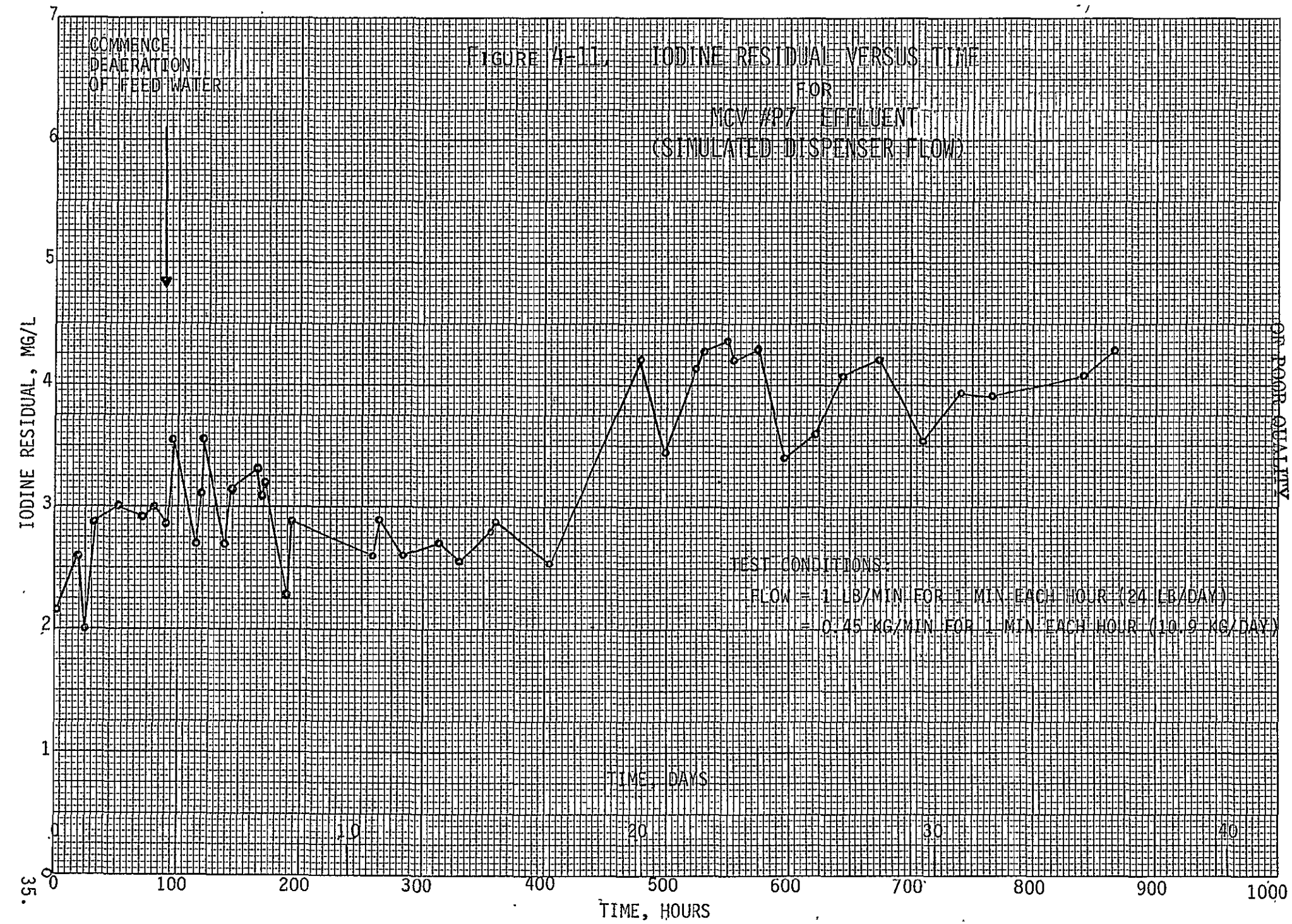

FIGURE 4-11. IODINE RESIDUAL VERSUS TIME FOR MCV #P7 EFFLUENT (SIMULATED DISPENSER FLOW)

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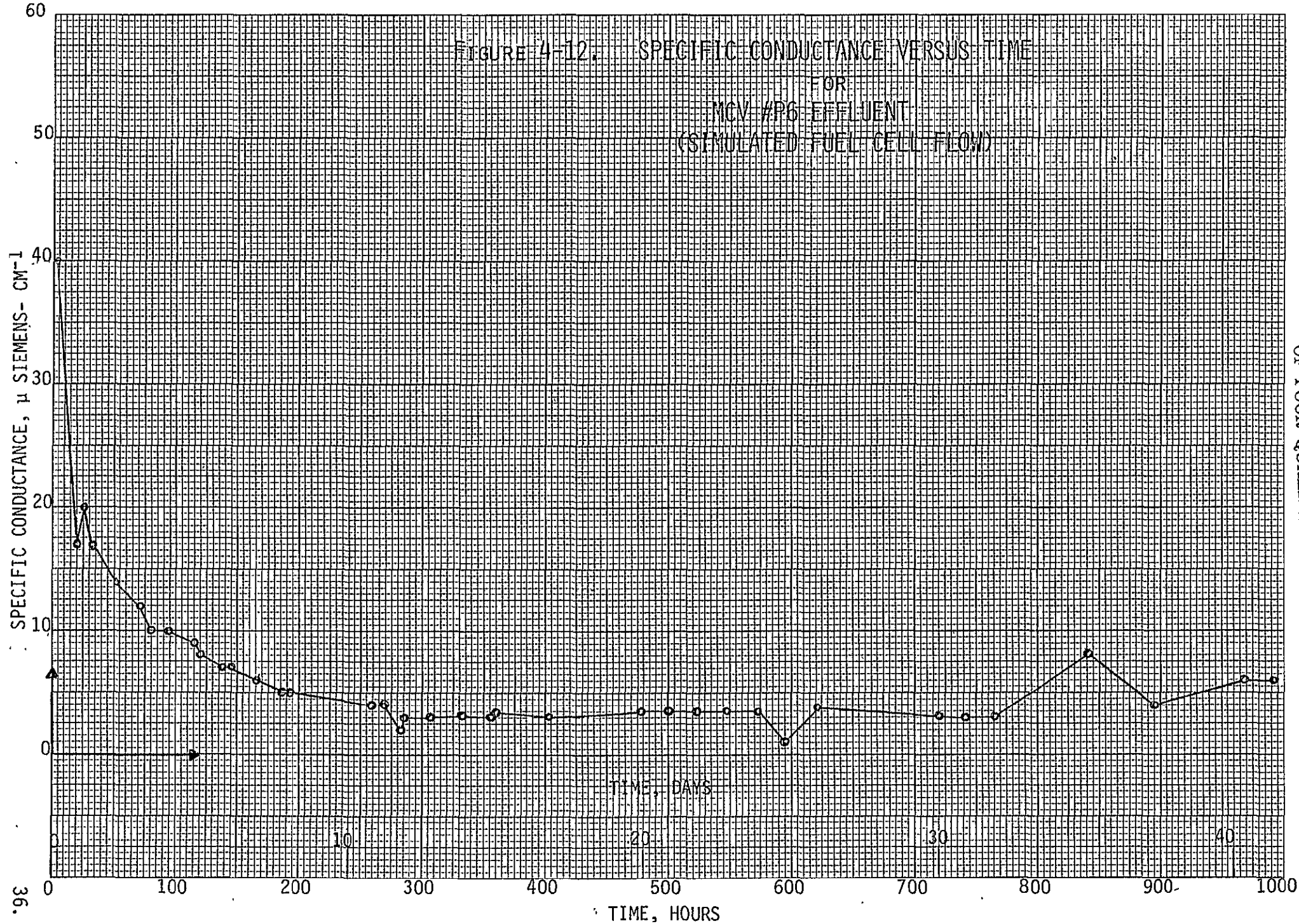

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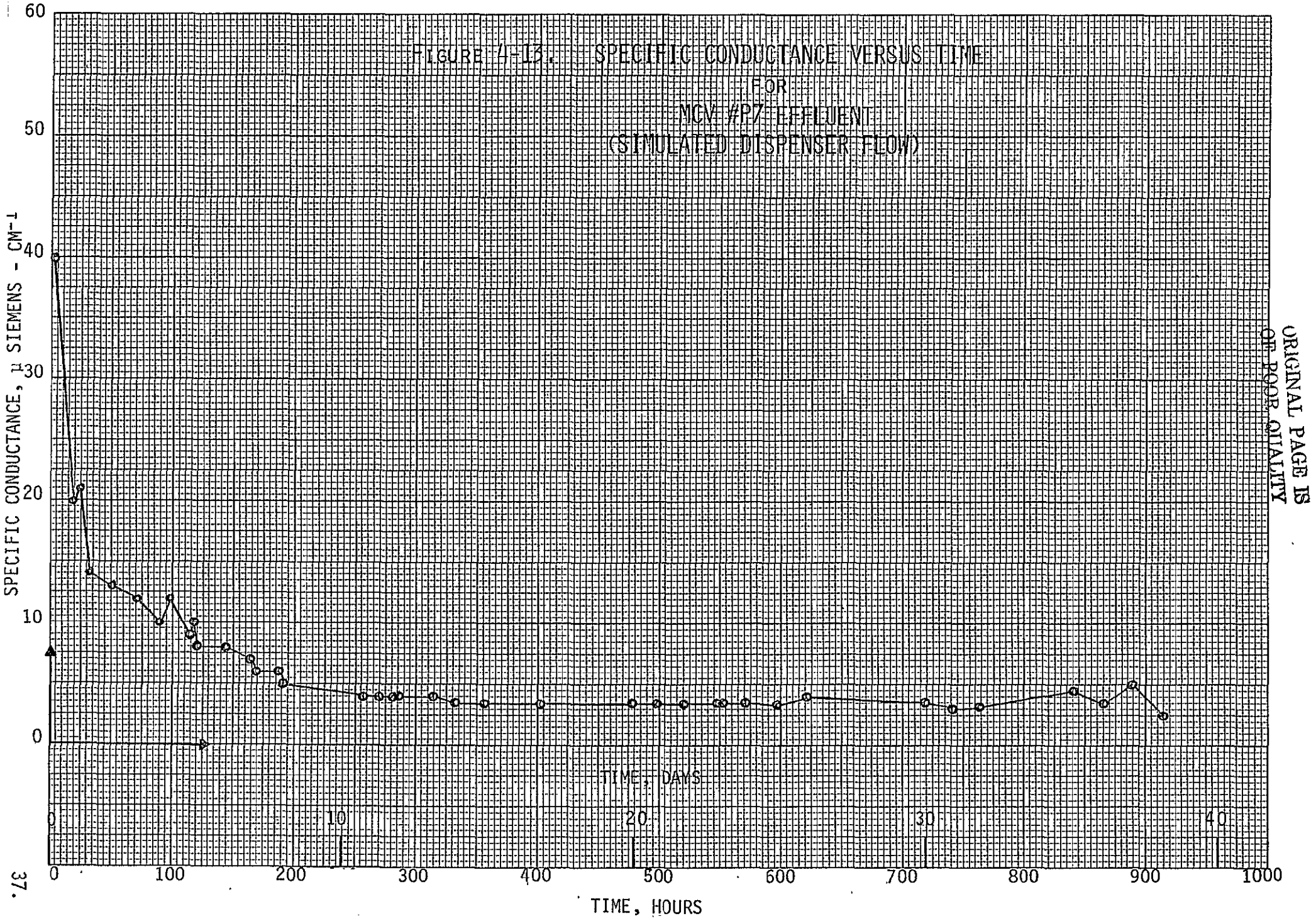

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4.6.2.7 pH. The effluent pH from both MCV units remained in the range 5 to 6 pH units with an average near 5.5 (See Figures 4-14 and 4-15). This is typical of previous tests.

This pH is somewhat below the specified range for the pH of Shuttle fuel cell water, which is 6 to 8 at 25°C. The low specific conductance of this water indicates that its total acidity is very low, thus pH associated corrosion, taste and health effects should be negligable.

4.6.2.8 Water Quality. Samples of the effluents from the two test beds were collected near the beginning, midpoint and end of the 30 day test. These data together with an analysis of the feed water are presented in Table 4-2.

The major impacts on the water quality are increased Odor and Color numbers introduced by the residual iodine. Otherwise, the units do not affect the water quality in a manner which impacts the Shuttle Water Quality requirements.

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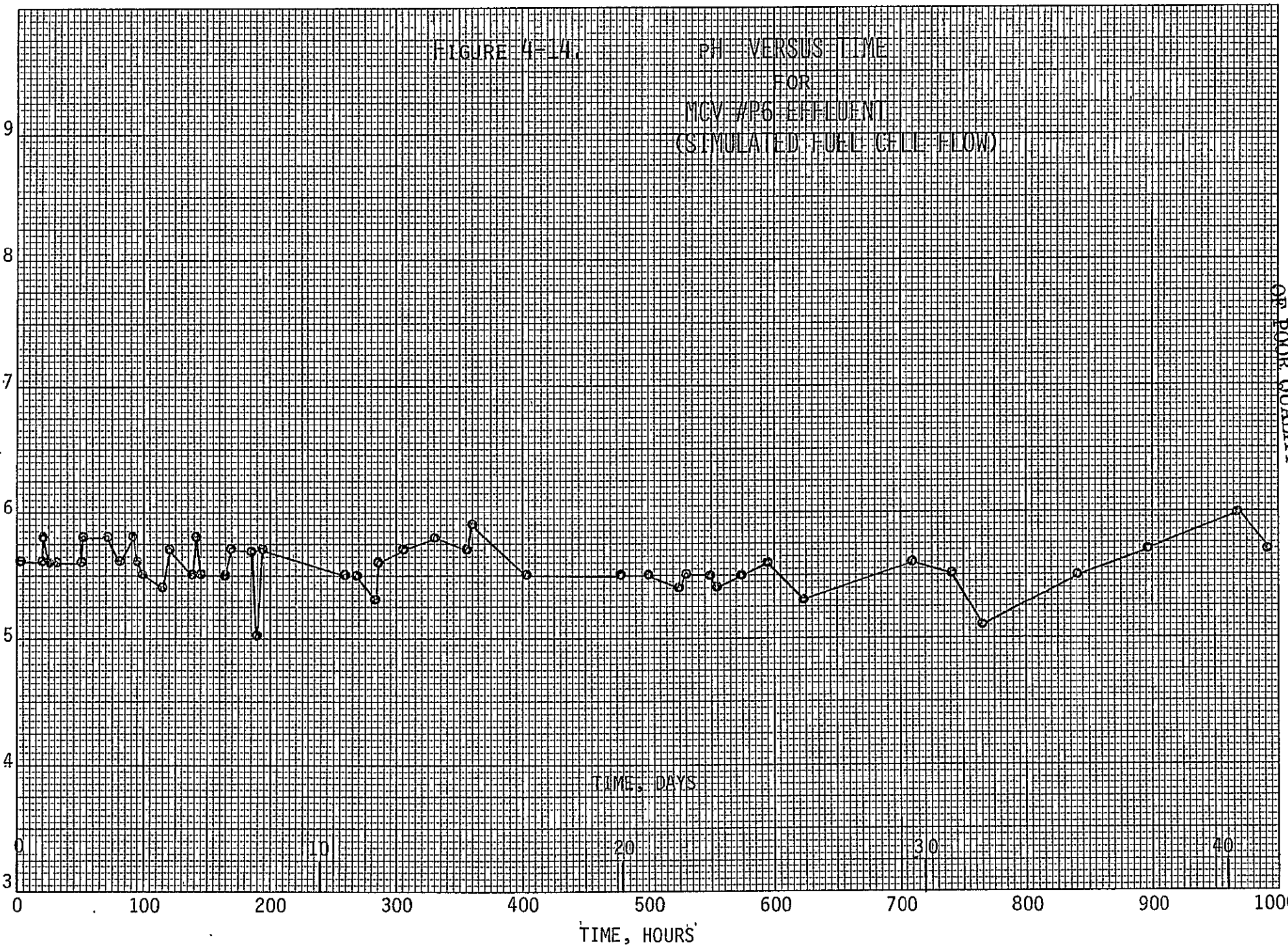
FIGURE 4-14. PH VERSUS TIME FOR MCV #P6 EFFLUENT (STIMULATED FUEL CELL FLOW)



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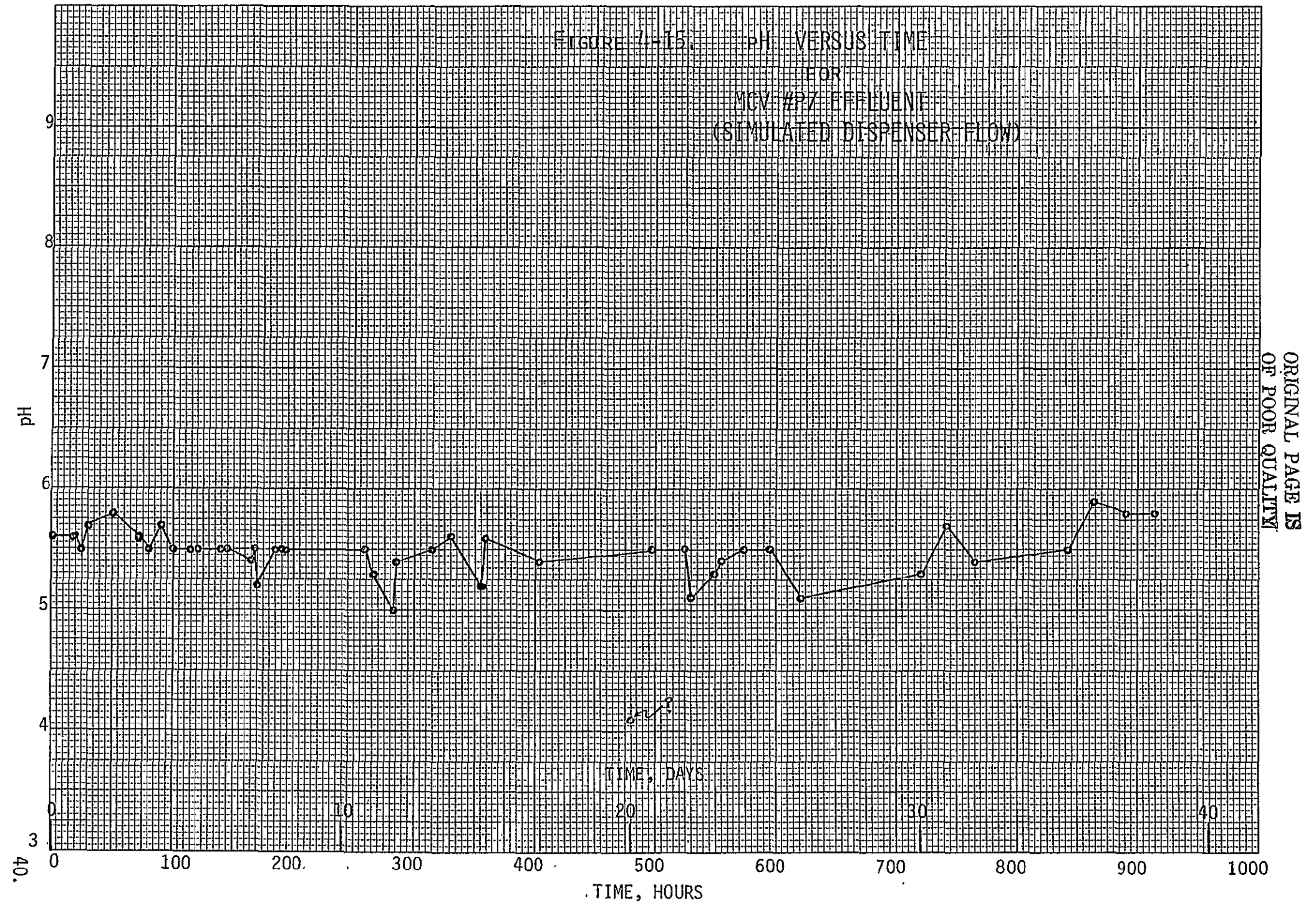

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TABLE 4-2, WATER QUALITY DATA

TEST	Test Day	2		12		36		
	Bed Units	P6	P7	P6	P7	P6	P7	Influent
pH	pH Units	5.7	5.6	5.6	5.4	5.7	5.9	5.7
Spec. Cond.	µmho/cm	22	21	3.0	4.0	3.9	3.5	3.0
Total Solids	mg/l	31	-	-	<1	<1	12	<1
T.O.C.	mg/l	1.5	-	-	1.1	0.1	0.03	<1
Odor	T.O.N.	3	-	-	3	2	2	<1
Turbidity	TU	<1	-	-	<1	<1	<1	<1
Color, True		25	-	-	25	15	30	<5
Cadmium	mg/l	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Chromium	mg/l	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Copper	mg/l	0.04	0.12	0.02	<0.01	0.02	<0.02	<0.01
Iron	mg/l	0.05	0.27	0.2	0.1	0.57	0.4	0.17
Lead	mg/l	0.007	0.01	0.001	<0.001	<0.001	<0.001	<0.001
Manganese	mg/l	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Mercury	mg/l	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Nickel	mg/l	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Silver	mg/l	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Zinc	mg/l	0.07	0.11	0.05	0.03	0.07	0.05	0.04
Selenium	mg/l	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002

4.7 DEVELOPMENT OF A RESIN CERTIFICATION TEST

Two procedures were developed to measure the iodine content of prepared resins.

4.7.1 Iodine Titration

A premeasured quantity of resin is soaked in a standard solution of sodium thiosulfate for 48 hours. The active iodine is leached from the resin and neutralizes part of the thiosulfate. The solution is then decanted from the resin and back titrated with standard iodine. The iodine content is then calculated. This procedure is applicable to all resin preparations.

4.7.2 Dry Density Relationship

The dry density of prepared resisn is not only correlatable with iodine content but also with the initial equilibrium residual iodine output with the higher residual resin. This correlation is shown in Figure

4.7.3 Certified Process Specification

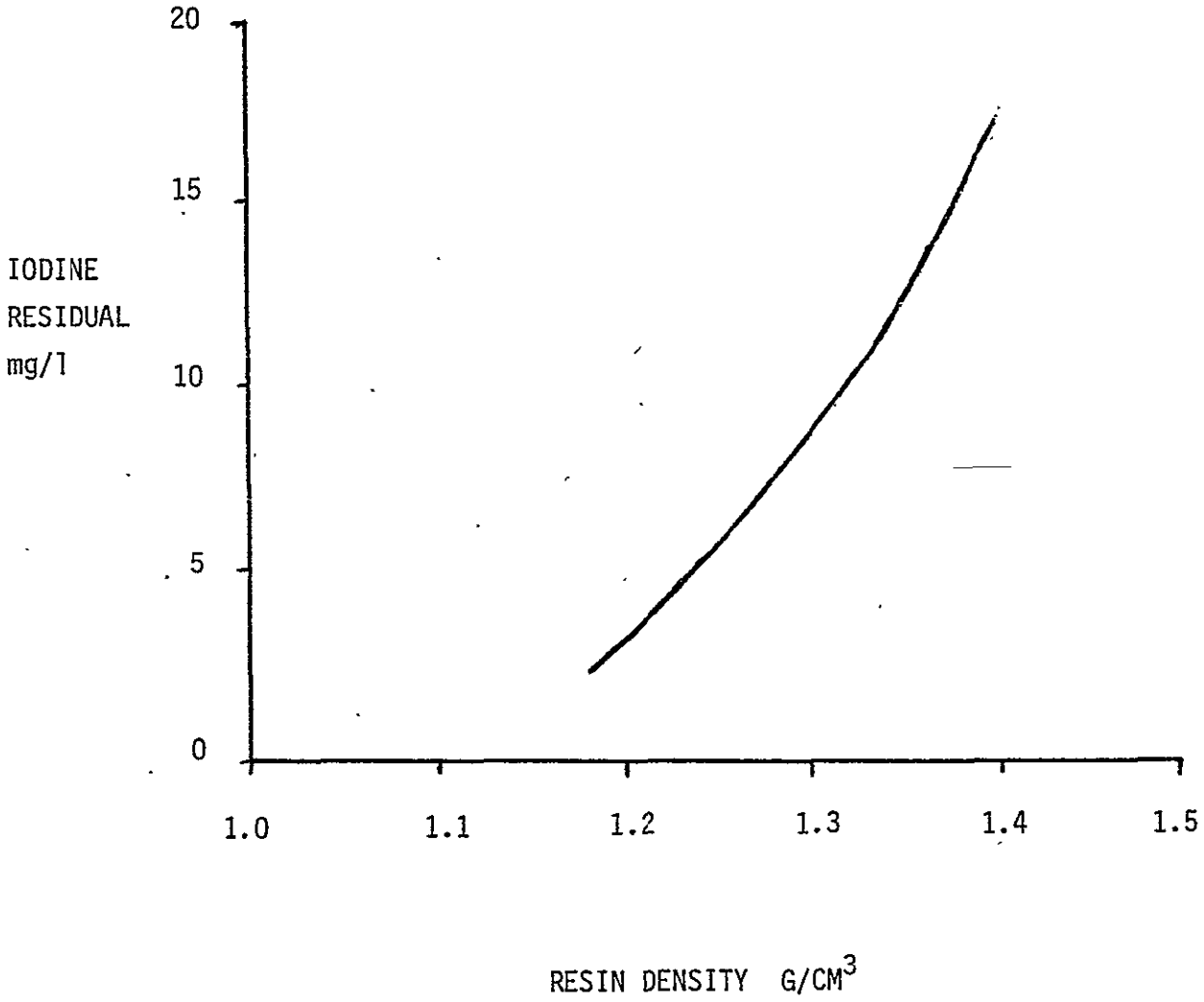
A process specification for the preparation of resin certified for use in the Space Shuttle program has been prepared. Although the above titration and density correlations are valuable for the estimation of resin iodine content, the certification procedure calls for measurement of the iodine residual under controlled conditions similar to mission requirements, to provide assurances that each batch will perform according to required guideline.

Certified Process Specification CPS 100 Follows.

5.0 End Item Hardware

The Microbial Check Valve Protoype Part No. MCVP-02, Serial No's P1, P2, P3 and P4 were shipped to Transportation Officer, NASA/ Lyndon B. Johnson Space Center. Serial No. P5 was shipped to Life Systems, Inc. Cleveland, Ohio. Copies of form DD250 are included.

Figure 4-16. RESIN DENSITY VS IODINE RESIDUAL



MATERIAL INSPECTION AND RECEIVING REPORT

1. PROC. INSTRUMENT IDEN (CONTRACT)	(ORDER) NO.	6. INVOICE	7. PAGE	OF
Contract NAS9-15079		NO.	1	1
Purchase Request 6-012-008		DATE	8. ACCEPTANCE POINT	

2. SHIPMENT NO.	3. DATE SHIPPED	4. D/L	5. DISCOUNT TERMS
1	5-12-78	TCN	

9. PRIME CONTRACTOR	CODE	10. ADMINISTERED BY	CODE
UMPQUA RESEARCH COMPANY P. O. BOX 791 MYRTLE CREEK, OR 97457			

11. SHIPPED FROM (If other than 9)	CODE	FOB:	12. PAYMENT WILL BE MADE BY	CODE

13. SHIPPED TO	CODE	14. MARKED FOR	CODE
Transportation Officer, Bldg. 420 NASA/Lyndon B. Johnson Space Center Houston, TX 77058		Accountable Property Officer 807402 For Reissue to: Richard L. Sauer DE5 Bldg. 36 Room 106	

16. ITEM NO.	15. STOCK/PART NO. DESCRIPTION (Indicate number of shipping containers - type of container - container number.)	17. QUANTITY SHIP/REC'D*	18. UNIT	19. UNIT PRICE	20. AMOUNT
1 thru 4	Microbial Check Valve Prototype Part No. MCVP-02 Serial No's P1,P2,P3,P4	4		2500.00	10,000.00
5	Collar	1		50.00	50.00

21. PROCUREMENT QUALITY ASSURANCE

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☐ PQA ☐ ACCEPTANCE of listed items has been made by me or under my supervision and they conform to contract, except as noted herein or on supporting documents.

DATE _____ SIGNATURE OF AUTH GOVT REP _____

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13. SHIPPED TO CODE:
Life Systems, Inc.
24755 Highpoint Road
Cleveland, Ohio 44122

14. MARKED FOR CODE:

15. ITEM NO.	16. STOCK/PART NO. (Indicate number of shipping containers - type of container - container number.) DESCRIPTION	17. QUANTITY SHIP/REC'D*	18. UNIT	19. UNIT PRICE	20. AMOUNT
1	Microbial Check Valve Prototype Part No. MCVP-02 Serial Number P5	1		2500.00	2500.00

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22. RECEIVER'S USE
Quantities shown in column 17 were received in apparent good condition except as noted.
DATE / SIGNATURE OF AUTH GOVT REP
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APPENDIX A.

IODINATED RESIN PREPARATION - SP102

UMPQUA RESEARCH CO.	NO. SP102	REV.
STANDARD PROCEDURES	DATE	Page 1 Of
TITLE: IODINATED RESIN PREPARATION (2 ppm Residual Type)	Prepared By: Charles L. Casad	
	Approved By:	

1.0 SCOPE

This document describes procedures for making Iodinated Resin which will exhibit a 2 ppm iodine residual when used in the Space Shuttle Microbial Check Valve as specified in Certified Process Specification CPS 100.

The quantities listed here will produce 800 grams of finished resin. If a different batch size is required, the quantities may be adjusted proportionally.

2.0 MATERIALS

		Record Lot Numbers
Iodine Crystals	- Amachem U.S.P. Resublimed	_____
Potassium Iodide	- Amachem U.S.P.	_____
Resin	- Ionac A-544, 16 to 50 mesh	_____
Water	- Deionized, Filtered (Whatman #41 filter).	
	- Record Specific Conductance	_____

3.0 EQUIPMENT

Triple Beam Balance - Ohaus 750-S

Standard Laboratory equipment.

4.0 RESIN STRIPPING

a. Weigh 686 ± 5 grams IONAC A-544 resin into a glass container.

Record resin wt._____

b. Weigh 686 ± 5 grams of Potassium Iodide, KI, into a glass container with a 7.5 l or greater capacity.

Record KI wt._____

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4.0 (cont)

c. Add 6.9 $\pm .1$ l Deionized water to KI and stir until KI is dissolved.

Record water volume_____

d. Add resin from (a) to the KI solution and stir at about 200 rpm with an overhead stirrer for 2 $\pm \frac{1}{2}$ hours.

Record stirring time_____

e. Decant the solution from the resin, add 2 l D.I. water, shake well, decant water add 2 l D.I. water, stir well, and let stand for 1 $\pm \frac{1}{2}$ hours.

Record standing time(1)_____

f. Repeat step e. hourly for 4 hours, 5 total rinses.

Record standing time (2)_____

Record standing time (3)_____

Record standing time (4)_____

Record standing time (5)_____

5.0 RESIN IODINATION

a. Dissolve 990 ± 10 grams KI in 10 ± 0.5 l filtered, deionized water.

Record KI wt._____

Record water volume_____

b. Add 432 ± 2 grams Iodine crystals and stir with overhead stirrer until Iodine is completely disolved.

Record Iodine wt._____

NOTE: Use extreme caution in handling Iodine crystals.

c. Add the resin prepared in par. 4.0 to the KI-I_2 solution prepared in steps a and b and stir with the overhead stirrer for 2 hours $\pm$ 5 min. at 200 rpm. Let stand for 18 ± 2 hours.

Record stirring time_____

Record standing time_____

d. Decant the solution from the resin, add 2 l deionized water, shake well, decant water add 2 l D.I. water, stir well, and let stand for $1^{\pm\frac{1}{2}}$ hours. Use filtered D.I. H_2O.

Record standing time (1)_____

e. Repeat step d. hourly for 4 hours, 5 total rinses.

Record standing time (2)_____
Record standing time (3)_____
Record standing time (4)_____
Record standing time (5)_____

6.0 RESIN DRYING

Spread resin evenly on a glass sheet and place in evenly distributed low heat source at $323^{\pm 10}$ K (122± 180F) in a well ventilated fume hood.

NOTE: Iodine fumes are very toxic. Do not allow fumes to escape into the laboratory from the fume hood.

7.0 RESIN STORAGE

Store the dry iodinated resin in clean glass jars with enameled lids. record Batch No. and date sealed on the Use Record Form F105 and attach it to the jar. Log the jar of resin into the storage area on the Storage Area Log Form F106.

Record Batch No._____
Date Stored_____